

**COAL CLEANING USING RESONANCE DISINTEGRATION
FOR MERCURY AND SULFUR REDUCTION PRIOR TO
COMBUSTION**

FINAL REPORT

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Centennial, Colorado**

**And
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National Energy Technology Laboratory
Morgantown, West Virginia**

**By
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Laramie, Wyoming**

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ABSTRACT

Coal-cleaning processes have been utilized to increase the heating value of coal by extracting ash-forming minerals in the coal. These processes involve the crushing or grinding of raw coal followed by physical separation processes, taking advantage of the density difference between carbonaceous particles and mineral particles. In addition to the desired increase in the heating value of coal, a significant reduction of the sulfur content of the coal fed to a combustion unit is effected by the removal of pyrite and other sulfides found in the mineral matter.

WRI is assisting PulseWave to develop an alternate, more efficient method of liberating and separating the undesirable mineral matter from the carbonaceous matter in coal. The approach is based on PulseWave's patented resonance disintegration technology that reduces that particle size of materials by application of destructive resonance, shock waves, and vortex generating forces.

Illinois #5 coal, a Wyodak coal, and a Pittsburgh #8 coal were processed using the resonance disintegration apparatus then subjected to conventional density separations. Initial microscopic results indicate that up to 90% of the pyrite could be liberated from the coal in the machine, but limitations in the density separations reduced overall effectiveness of contaminant removal. Approximately 30-80% of the pyritic sulfur and 30-50% of the mercury was removed from the coal. The three coals (both with and without the pyritic phase separated out) were tested in WRI's 250,000 Btu/hr Combustion Test Facility, designed to replicate a coal-fired utility boiler. The flue gases were characterized for elemental, particle bound, and total mercury in addition to sulfur. The results indicated that pre-combustion cleaning could reduce a large fraction of the mercury emissions.

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EXECUTIVE SUMMARY

In 2004 PulseWave LLC and Western Research Institute (WRI) conducted a jointly sponsored research project to evaluate PulseWave's resonance disintegration technology as a method for pre-combustion removal of mercury and sulfur from coal. The technology allows for high efficiency liberation of pyrite from coal during pulverization. Combined with an appropriate separation technology, this approach has the potential to significantly reduce the emission of mercury, sulfur, and other toxic compound during combustion of coal.

During the project, large samples of Wyodak, Illinois #5, and Pittsburgh #8 coals were obtained and pulverized in PulseWave's apparatus at their facility in Centennial, Colorado. Approximately half of each pulverized sample was sent to a commercial vendor for separation of the pyritic and carbonaceous phases. Each of the pulverized, and pulverized/cleaned coals was then characterized with proximate, ultimate, forms of sulfur, and mercury analyses. The tests showed that 82% of the pyritic sulfur was removed from the Wyodak coal, while 26% of the pyritic sulfur was removed from the Illinois#5 coal, and 20% of the pyritic sulfur was removed from the Pittsburgh #8 coal. The process removed 30% of the mercury from the Wyodak coal, and 14% of the mercury from the Pittsburgh coal, but the tests indicated that the Illinois#5 coal was contaminated with mercury from an external source.

The samples were then sent to WRI's facilities in Laramie, WY for tests in a pilot-scale Combustion Test Facility (CTF). Combustion flue gases were monitored with on-line analyzers for NO_x and sulfur emissions. A subcontractor was hired to monitor mercury emissions by the Ontario-Hydro method. Again, the results indicated that the Illinois coal was contaminated from an external source. Reductions in mercury emissions for the Wyodak and Pittsburgh coals were much higher than expected, with 50% less emissions for the cleaned Wyodak coal and 90% less emissions for the Pittsburgh coal.

The results indicated that the PulseWave technology is a viable part of a pre-combustion removal of mercury and pyritic sulfur from coal. Further development may be needed in the separation of the pyrite from the coal.

INTRODUCTION

About one half of the coal mined in the U.S is cleaned to some extent; for Eastern coals the figure may increase to about three fourths. The purpose of coal cleaning is to simply modify raw coal into a more preferred form before it is used. These processes involve the crushing or grinding of raw coal followed by physical separation processes, taking advantage of the density difference between carbonaceous particles and mineral particles. In addition to the desired increase in the heating value of coal, a significant reduction of the sulfur content of the coal fed to a combustion unit is affected by the removal of pyrite (FeS_2) and other sulfides found in the mineral matter. Other potential sources of toxic emissions found in the mineral phase, such as mercury, selenium, arsenic, lead, nickel, manganese, and cadmium also can be removed pre-combustion. Mercury has become a toxic emission of particular concern and is extremely difficult and costly to remove from the post-combustion flue gas of power plants¹. In addition, mercury concentrations in flue gas tend to be extremely low.

The pre-combustion separation and removal of the mineral phase from coal has the potential to significantly reduce the post-combustion emissions of mercury, sulfur, and other potential toxic compounds.

BACKGROUND

The purpose of coal cleaning is to remove mineral matter and impurities and to increase the organic content of coal. Coal cleaning typically involves size reduction, particle sizing, cleaning, and dewatering. Most of the coal cleaned commercially is separated by density-based processes, which are particularly effective for removing pyrite².

The first step in most coal cleaning processes is size reduction, which serves to liberate mineral matter from carbonaceous matter. Size reduction of raw coal traditionally involves fracturing the coal in a crushing media such as a ball mill/pulverizer or by impacting coal-to-coal particles at high velocity as in a jet mill. Such processes are limited as to the potential minimum size that can be achieved without “smearing” together the carbonaceous and mineral phases, resulting in substantial carry-over of minerals with the coal through a separator into the combustion unit, and conversely losing a fraction of the coal in the mineral matter.

Following size reduction, a variety of separation methods are utilized to remove undesirable mineral matter including: a Baum Jig, a dense-medium washer, a dense-medium cyclone, a hydrocyclone, froth floatation, and oil agglomeration. Coarser fractions are typically easier and less expensive to separate mineral matter; difficulty and costs increase for finer fractions³. Enhanced gravity separators such as a Falcon

Concentrator have been recently evaluated for fine coal cleaning with generally positive results^{4, 5}.

The purpose of this project is to evaluate the use of an alternate, more efficient method of liberating and then separating the undesirable mineral matter from the carbonaceous matter in coal. PulseWave, LLC has developed a patented resonance disintegration technology that reduces the particle size of materials by application of the physics of destructive resonance, shock waves, and vortex-generated shearing forces.

The PulseWave apparatus is a mechanical device designed to reduce the size of material composed of wet or dry discrete objects into relatively smaller particles with shock waves created by flowing the material through a housing having alternating rotors and orifice plates. The top of the housing includes a feed chute for introducing material, and an exit chute at the bottom for removing pulverized material. Inside the housing multiple rotors are mounted on a longitudinal shaft with orifices between them. The rotation of the rotors combined with the placement of the orifices causes a shock wave that causes material to break into smaller particles. Figure 1 shows a side view of the apparatus. Items 90, 92, 94, 96, 98, and 100 are the rotors. The orifice plates are items 128, 130, 132, 134, 136. Material flowing through an orifice in the pulverizer first undergoes a velocity increase and an accompanying decrease in pressure. Then, because the available volume decreases at each succeeding stage, the material experiences a rapid compression, which in turn can cause a rapid increase in pressure and/or temperature. The size of the orifice is increased with each succeeding stage to provide a pressure immediately downstream of an orifice that is lower than the pressure immediately upstream the orifice. This negative backpressure that is maintained across each orifice helps to maintain the flow.

The pulverizer has been tested with several different feedstock materials of widely varying composition, hardness, ductility, and moisture content. The results of the tests indicated that material introduced into pulverizer with rotor assembly spinning at speeds of approximately 1000 rpm or greater are pulverized primarily by shock waves generated within the housing. Observations indicated that material fed into the feed chute, as well as air entering through the feed chute, are accelerated rapidly and then entrained into a fluid-like flow through the spinning rotor assembly. It appears that the material in the flow is almost immediately subjected to a rapid-fire succession of shock waves, which may begin to break up the feedstock even before it reaches the distributor rotor.

The spinning rotors create a very strong airflow through the housing. It appears that material fed into the pulverizer is entrained in this flow. The material apparently flows with the air through the pulverizer making minimal contact with the sides of the housing or with the orifice plates. This is likely due to the flow being influenced by the

Coanda effect to closely follow the contours of the rotor peripheries and orifice rims. The Coanda Effect was discovered in 1930 by the Romanian aerodynamicist Henri-Marie Coanda (1885-1972) who observed that a stream of air (or a other fluid) emerging from a nozzle tends to follow a nearby curved surface, if the curvature of the surface or angle the surface makes with the stream is not too sharp.

The Coanda effect helps to reduce high-angle contacts between the flowing material and the component parts of the pulverizer, and thereby reduce wear on these parts. The Coanda flow rapidly changes direction as it rounds the peripheral edge of each rotor and the rim of each orifice, alternating between a flow that is directed radially outward and a flow that is directed radially inward. The sizes of the orifices increase with each succeeding stage to maintain a negative backpressure throughout the rotor assembly, which helps to keep the velocity of air and particles sufficiently high to maintain the Coanda flow. This effect is the primary reason that the machine is able to disintegrate large particles into smaller particles with very little wear. Shock waves are generated each time the material experiences rapid acceleration. Additional shock waves are generated as the rotor vanes pass corners in the housing. These effects are primarily responsible for the size reduction that takes place in the pulverizer instead of direct contact between the feed material and the machine. A more detailed description is included in U.S. Patent 6,135,370.

This technology has the following advantages when compared with conventional crushing and grinding:

- Carbonaceous materials are fragmented in a relatively “nondestructive” manner from within rather than being crushed by the more “destructive” impact or grinding processes. Since the carbonaceous material and mineral material in coal will have different elasticities, they will tend to resonate at differing frequencies to the shockwaves in the PulseWave machine, resulting in a relatively clean liberation of these two phases. This eliminates the “smearing” of the different fractions that can take place in conventional mills and should result in particle size reduction accompanied by a cleaner separation of the carbonaceous and mineral phases and less crossover of unwanted materials in each phase.
- Significantly less energy is required for the work being done by the PulseWave machine when compared with the energy required for traditional impact/grinding processes for comparable particle size reduction.
- The particles produced by the PulseWave process are generally statistically closer, in terms of size and dimensions, than those produced by normal crushing methods, which could result in more uniform and predictable combustion characteristics.

The pre-combustion separation and removal of the mineral phase from coal, using the PulseWave technology, has the potential to significantly reduce the post-combustion emissions of mercury, sulfur, and other potential toxic compounds. This should result in a significant reduction in post-combustion emissions control costs. Studies have indicated that for many coals including Pittsburgh #8 and Illinois #6 (note that Illinois #5 is tested here), mercury and many other toxic emissions were shown to correlate very strongly with pyritic sulfur⁶.

OBJECTIVES

In 2004 PulseWave LLC and Western Research Institute conducted a project to evaluate the effectiveness of applying the PulseWave resonance disintegration technology as a coal-cleaning process. The technology is designed to achieve a significant reduction in minerals and their associated toxic elements prior to combustion of coal. In particular, PulseWave is interested in the pre-combustion reduction of mercury and sulfur and the resultant effect on post-combustion emissions. Specific objectives for this project were to 1) demonstrate the effectiveness of PulseWave's process for removing toxic materials from coal prior to combustion, 2) assess the combustion and emissions characteristics of the cleaned coal in pilot scale combustion tests, and 3) assess the potential implications of incorporating the technology in a commercial scale coal fired power plant.

EXPERIMENTAL

Coal samples from Wyoming (Wyodak coal), Pennsylvania (Pittsburgh #8 coal) and Illinois (Illinois #5 coal) were obtained and processed in PulseWave's machine at their facility in Centennial Colorado.

After characterization for proximate/ultimate, forms of sulfur, and mercury by an independent laboratory, the samples were examined with photomicrographs to estimate the degree of liberation of pyrite from carbon in the coal.

Approximately 35 lb of each pulverized coal was then sent for separation of the pyrite and the carbonaceous phases. Initially, float-sink separation methods were examined, but the samples were finally separated using a commercially available separation process, which separates the mineral fraction from the pyritic fraction in water slurry. After separation, both the cleaned coal and the pyritic fraction were characterized again for proximate/ultimate, forms of sulfur, and mercury.

The samples were then sent to WRI's Combustion Test Facility (CTF). The CTF is a pilot-scale pulverized coal combustion facility located at WRI's Advanced Technology Center in Laramie, Wyoming (see Figure 2). The CTF is a 200,000 Btu/hr

balanced-draft system designed to closely replicate a pulverized coal-fired utility boiler. In its present configuration, the unit has been set up to simulate a tangential-fired boiler, but it may be easily adapted to wall-fired or other configurations. The fuel feed system consists of screw-based feeders and pneumatic transport to four burners inserted in the corners of a refractory-lined firebox. The unit is equipped with appropriately sized heat-recovery surfaces to replicate the time/temperature profile of a utility boiler.

These comprise a water-cooled waterwall section, and air-cooled super-heater, re-heater, and economizer simulators. The CTF also includes provisions for preheating the combustion air to mimic a utility air pre-heater, and over-fire air injection ports for combustion staging. The unit is equipped with a bag filter and solids and gas sampling. The gas analysis system includes on-line analyzers for the monitoring of O₂, NO_x, SO₂, CO, and CO₂. A subcontractor characterized off-gases for mercury emissions using the Ontario-Hydro method. Note that after these experiments were conducted, analyzers were added for elemental and oxidized vapor-phase mercury.

RESULTS

Because of the relationship between pyrite and trace elements (including mercury) in most coals, the primary focus of this work has been on the liberation and removal of pyrite with the intent of removing a large fraction of the mercury pre-combustion by removing a large fraction of the pyrite.

PulseWave Operating Information

Table 1 presents summary details of some of the PulseWave machine parameters, including energy consumption and an estimate of processing costs, for the three coals examined in this project.

The input size of the coals was approximately 1-3" in diameter, and the machine runs were all conducted at 4250-4350 rpm. The Wyodak coal was reduced to a d50 of 278 μ m after one pass through the machine and was run through a second time with a resulting d50 of 146 μ m. This second pass was deemed necessary not because of insufficient liberation, but because the large particles could have contained unexposed pyrite. It should be noted that the Wyodak is a sub-bituminous coal and is "softer" than either the Illinois or Pittsburgh bituminous coals, and this difference may have resulted in the larger particles after processing. The Illinois coal was similarly run twice, with respective d50's of 45 μ m and 33 μ m; after a microscopic examination of the results of both of these runs, it is felt that the degree of liberation did not significantly change, and that one run would be sufficient to liberate most of the pyrite. Based on this conclusion, the Pittsburgh coal was run only one pass through the machine, with a resulting d50 of 77 μ m.

Table 1. PulseWave Machine Operating Information for Coal Experiments

Coal	PulseWave Machine Pass#	d50 μm	Amps	Throughput Lb/hr	kW/ton	Cost/ton*
Wyodak	1	278	73	7000	13.8	\$1.11
	2	146	69	3655	25.1**	\$2.01**
Illinois#5	1	45	70	6200	15	\$1.20
	2	33	75	3429	29**	\$2.33**
Pittsburgh	1	77	72	7425	12.9	\$1.03

* - At \$0.08/kWh.

** - Cumulative total.

These costs, while not including separation costs (estimated at less than \$0.50 per ton), compare very favorably with conventional coal-cleaning costs.

Moisture Reduction

An unexpected benefit was noted in the coals after processing in the PulseWave apparatus. In each of the three coals processed, the moisture content was reduced from pulverization in the PulseWave apparatus. Figure 3 shows the moisture content of each coal before pulverizing, after pulverizing, and after the water-based separation of pyrite. Moisture reduction of over 2% (by weight) were observed for both the Wyodak coal and the Illinois coal after pulverization, although for the Illinois coal this is nearly 50% of the moisture that it started with. Both the Wyodak and the Pittsburgh coals regained a substantial amount of moisture from the separation process.

Pyrite Liberation and Separation

After preliminary microscopic examination, the processed coals were sent to the Maceral Separation Laboratory at Southern Illinois University (SIU) for examination. SIU noted that pyrite was present in the subject coals as small single crystals, framboids, and massive vein and cell fillings, and that based on petrographic analysis all of these types of pyrite are being liberated. SIU also estimated that approximately 90-95% of the pyrite was liberated from the carbonaceous fraction in all three coals. Figures 4-6 on the following pages show photomicrographs of each coal as received and after processing on the PulseWave apparatus.

Samples of the raw, unprocessed coals and the PulseWave processed coals were submitted for before-and-after analyses for proximate/ultimate, sulfur forms, total

mercury, and elemental mercury. Approximately half of the processed material was then run through a commercial separation process to remove the liberated pyrite from the organic material. The resulting cleaned coal and mineral phases were sent for analysis and tests in WRI's Combustion Test Facility to determine handling, feeding, combustion, and emissions characteristics. Although it was not an objective of the project, the cleaning process recovered a high percentage of the original BTU value for all three coals. For the Wyodak sample, 95% of the BTU value was retained in the cleaned fraction. 90% of the BTU value was retained in the Pittsburgh coal, and 98.5% of the BTU value was retained in the Illinois#5 coal.

To illustrate the degree of liberation of the pyrite from the organic coal matrix, SIU separated a sample of each processed coal into float (organics) and sink (pyrite) components in a Density Gradient Centrifuge (DGC) and took photomicrographs. Figures 7, 8 and 9 show microphotos of the pyrite from each coal and give an illustration of the degree to which the pyrite is cleanly liberated from the coal. Very little carbon is visible in the photomicrograph.

Figure 10 shows the pyritic sulfur concentrations in the processed coal and the "cleaned" coal fractions. The liberation and separation was effective for the Wyodak coal, with approximately 82% of the pyritic sulfur removed. The process was much less effective for both the Illinois #5 and the Pittsburgh #8 coals with approximately 26% and 20% of the pyritic sulfur removed respectively. The pyritic sulfur for the Illinois coal decreased 64% (compared to 26%) when the separation was performed by DGC, indicating that the separation process limited the removal. These observations are further supported by the photomicrographs of the cleaned coals in Figures 11-13. The photomicrograph of the cleaned Wyodak coal (Figure 11) shows a small amount of un-liberated pyrite, but very little liberated but un-separated pyrite. The photomicrographs of the cleaned Illinois coal and the cleaned Pittsburgh coal (Figures 12 and 13 respectively) show a substantial amount of liberated but un-separated pyrite. The residual pyrite appears to be predominantly very fine particles, usually less than 5 microns in diameter.

Mercury Reduction

As mentioned above, one of the goals of this research was to remove mercury by removing a portion of the pyrite from the subject coals. To verify this, total mercury of each coal was measured in the raw samples and then in the cleaned samples. The results for the Wyodak coal and the Pittsburgh coal are shown in Figure 14 below. Approximately 30% of the mercury was removed with the pyrite in the Wyodak coal, but only 14% of the mercury was removed from the Pittsburgh coal. These reductions are substantially less than the reductions measured for pyritic sulfur, indicating that there may be a substantial portion of the mercury present in organic form not associated with

the pyrite. The reduction of mercury for the Pittsburgh #8 coal is in the range reported in a study by Consol Inc⁷, which reported coal-cleaning studies for several unidentified Pittsburgh seam coals.

Figure 15 show the mercury concentrations in the Illinois #5 coal before and after removal of the pyrite. At first glance it appears that the pulverizing and cleaning process has concentrated the mercury in the carbonaceous phase. A study by CQ Inc indicated the possibility of concentration of mercury in “cleaned” coal for some types of separations processes⁸; however, a material balance between for mercury between the “cleaned” phase, the pyrite phase, and the pulverized coal resulted in a recovery of 340% of the mercury in the pulverized coal. The material balance clearly indicates that the Illinois “cleaned” coal was contaminated from an external source. The balances for the Wyodak coal and the Pittsburgh coal were 88% and 106% respectively. PulseWave indicated that for the Illinois coal, an ore sample processed earlier in the separation apparatus might have contaminated the sample (even though it is cleaned between each material). This theory is further supported by the results of the combustion tests for the Illinois coal in Figure 16. The “cleaned” coal had nearly quadruple the mercury emissions of the raw pulverized coal. By contrast Figures 17 and 18 illustrate the reduction in mercury emissions during the combustion tests for the Illinois and Pittsburgh coals. The reduction in mercury emissions during the combustion tests greatly exceeded the reductions measured from the coal characterization samples, with over 50% less emissions in the cleaned Wyodak coal than the pulverized coal and over 90% less emissions in the cleaned Pittsburgh coal than the pulverized coal (as measured by the Ontario-Hydro method). It appears likely that the coal cleaning process reduces forms of mercury not removed by the baghouse on the CTF.

CONCLUSIONS

1. The PulseWave Apparatus appears to be very effective at liberating pyrite from carbon in all three coals tested.
2. Pulverizing coals in the PulseWave machine reduces the moisture content.
3. The effectiveness of pyrite removal varies for the different coals.
4. The removal of pyritic sulfur was limited by the effectiveness of the separation process employed.
5. A substantial portion of the mercury was removed with the pyrite in the Wyodak coal and the Pittsburgh coal.
6. Reductions in mercury emissions were much higher in combustions tests for the Wyodak and Pittsburgh coals than what was expected from coal analyses.

RECOMMENDATIONS

1. A key to potential commercial application of the PulseWave technology will be to improve the separation between the pyritic and carbonaceous phases. A dry separation process would take advantage of the reduction in moisture during pulverization.
2. Additional coals from other sites should be tested.
3. Future tests should include measurement of other trace contaminants of interest including arsenic, lead, and chromium.

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WRI Combustion Test Facility (CTF)

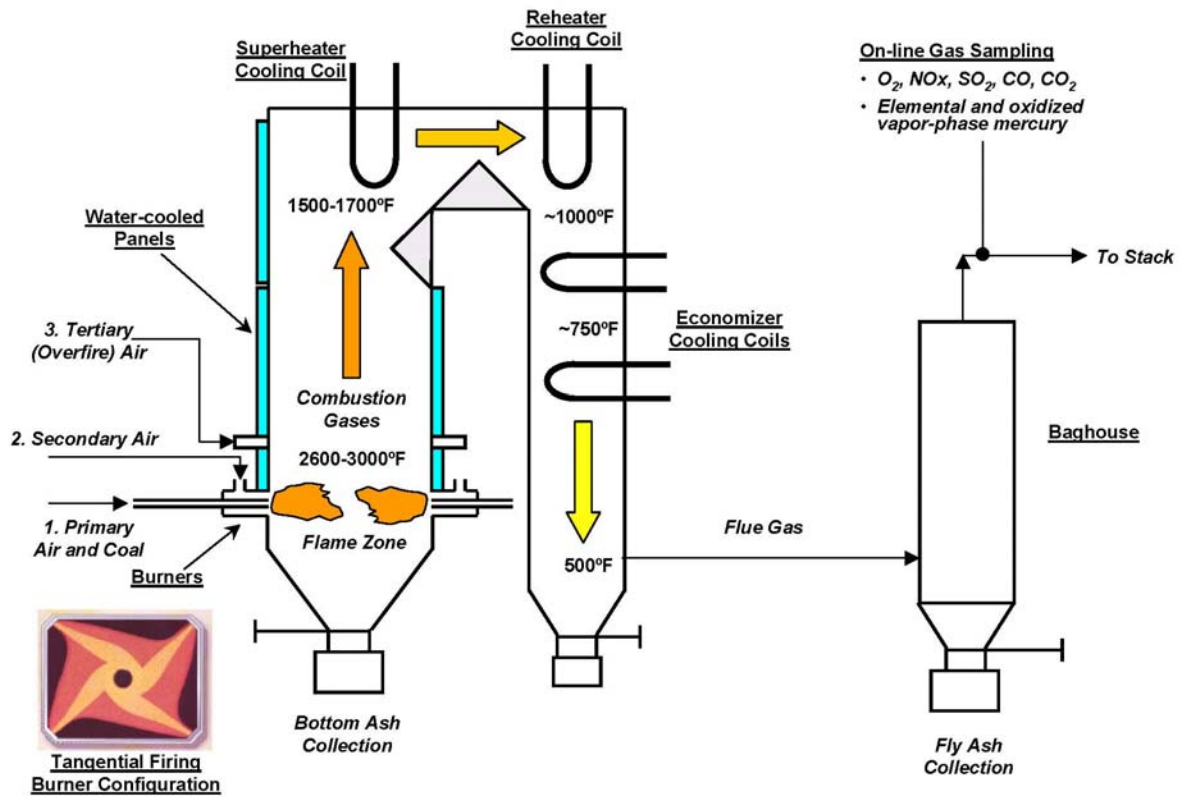


Figure 2. Schematic of Combustion Test Facility at WRI

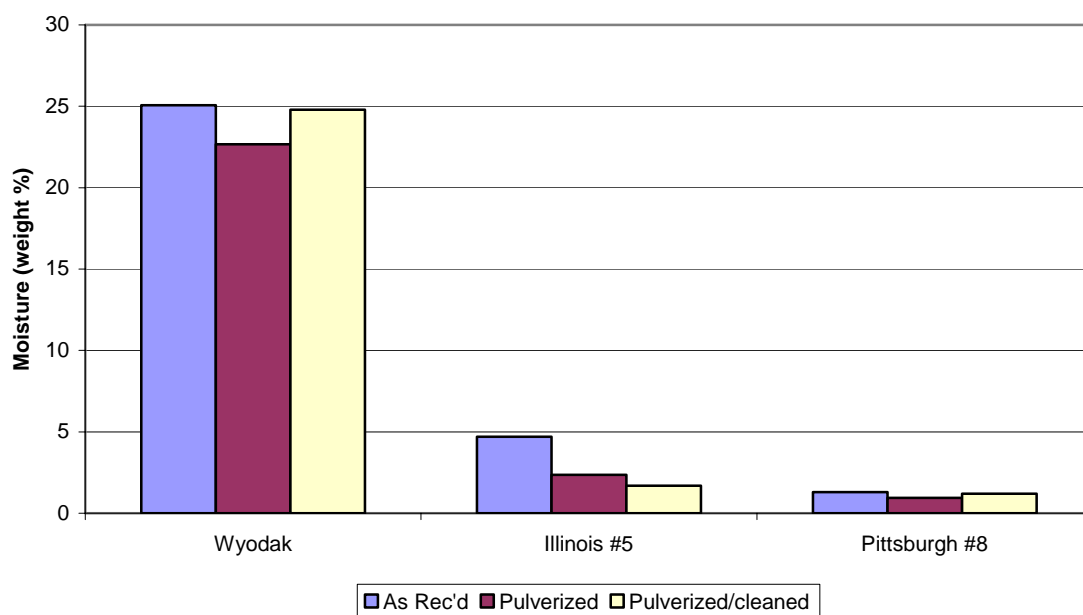


Figure 3. Moisture content of Wyodak, Illinois#5, and Pittsburgh #8 coals processed in the PulseWave machine.

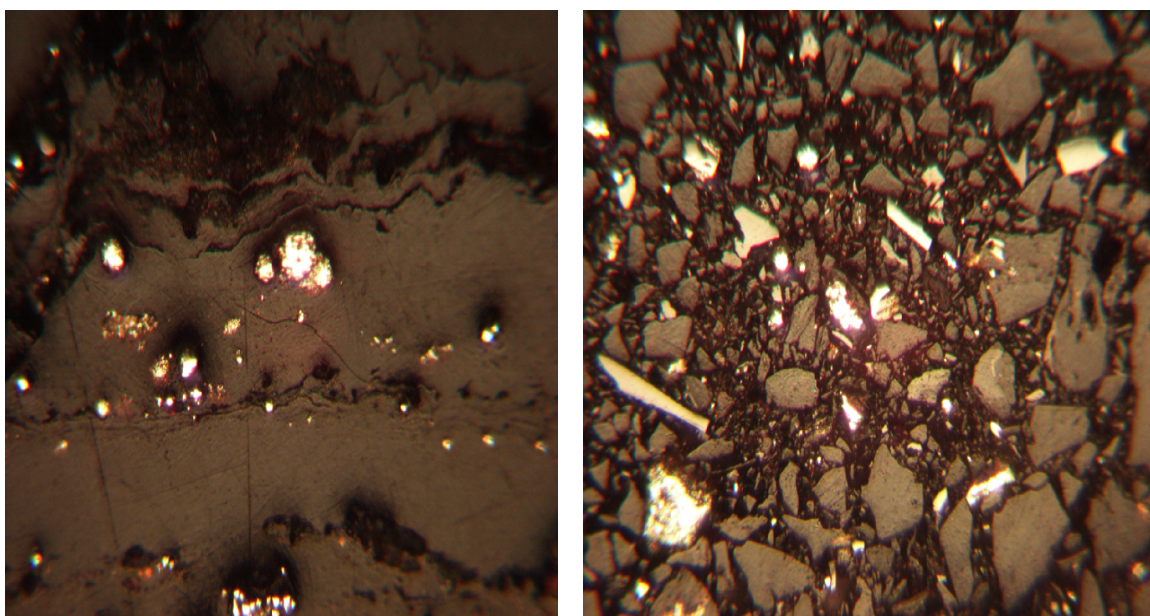


Figure 4 Photomicrographs (~ 450x) of a raw sample of the Illinois #5 coal (left panel) showing *in situ* pyrite crystals and framboids and a sample of the same coal after processing through the PulseWave machine (right).

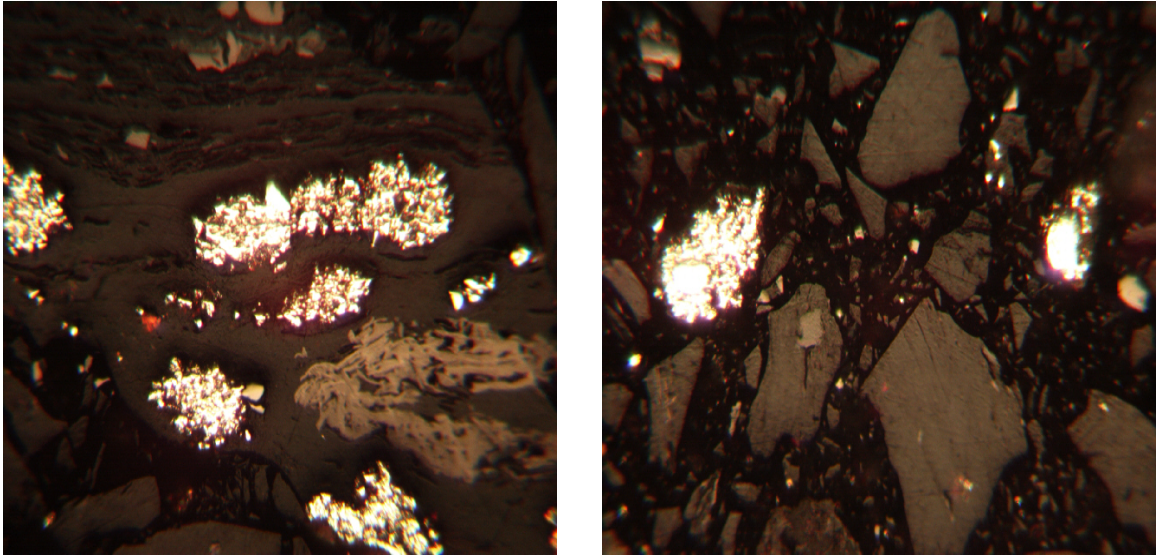


Figure 5. Photomicrographs (~ 450x) of the Pittsburgh #8 coal (left panel) showing large pyrite framboids and smaller crystals in place in the raw coal. Pulverized sample (right panel) showing liberation of both the large framboids and crystals.

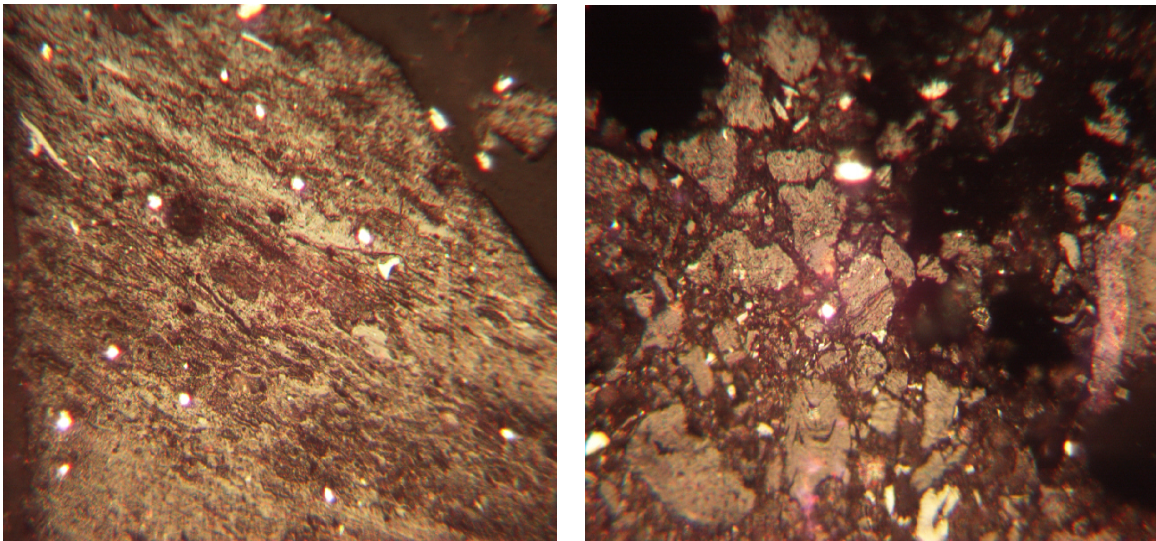


Figure 6. Photomicrographs (~ 450x) of the Wyodak coal. Left panel is the raw coal with small pyrite crystals in place; right panel is processed Wyodak with liberated pyrite.

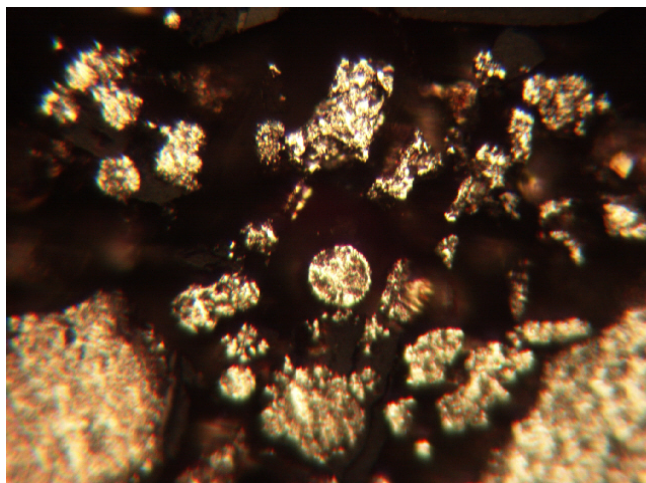


Figure 7. Photomicrograph of liberated and DGC-separated pyrite from the Illinois #5 coal.

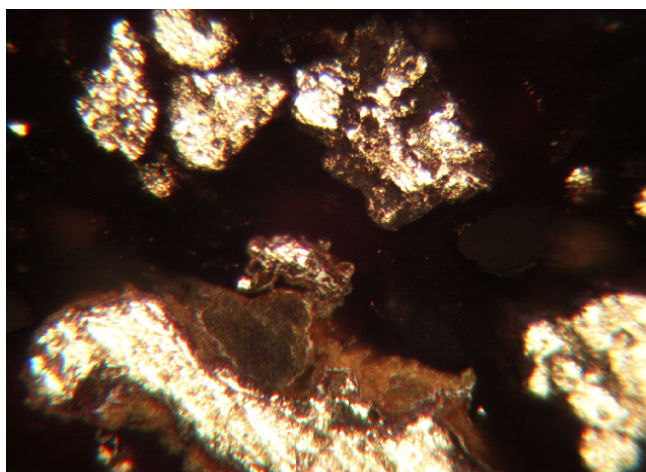


Figure 8. Liberated and separated pyrite from the Pittsburgh #8 coal.

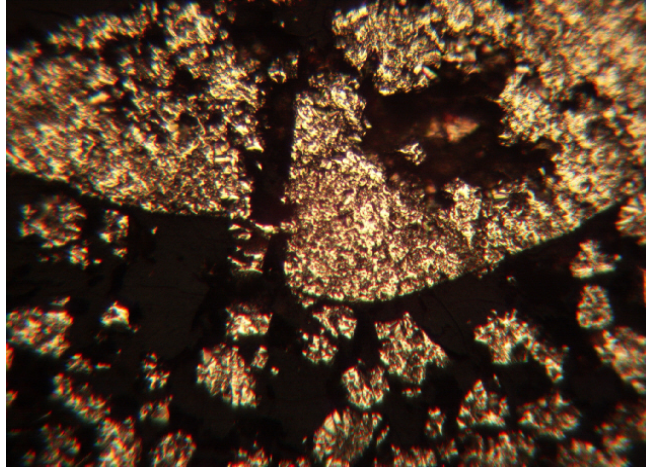


Figure 9. Photomicrograph of liberated and separated pyrite from the Wyodak coal.

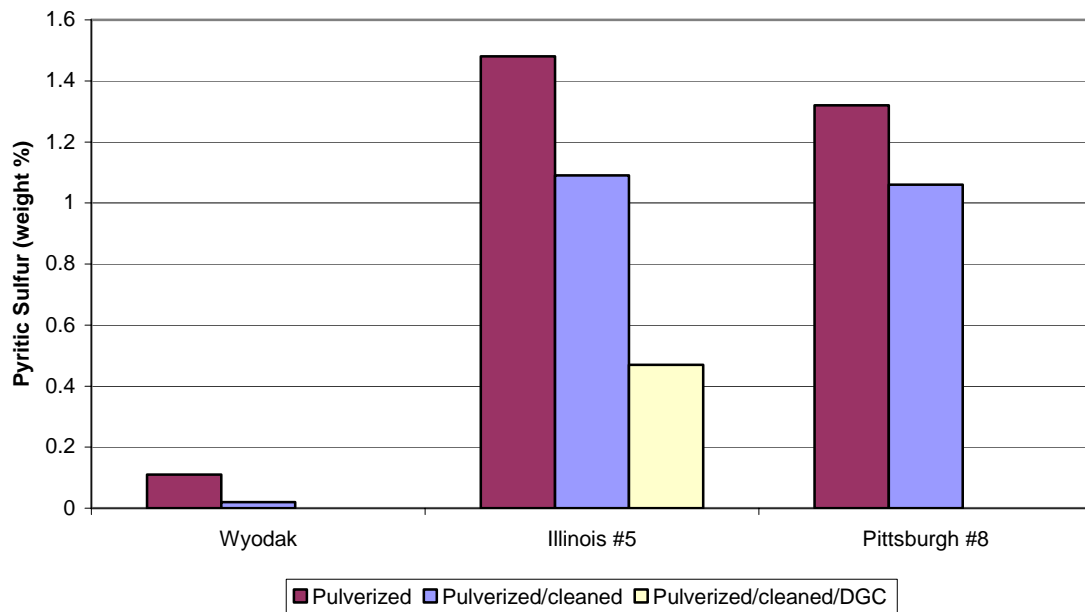


Figure 10. Pyritic Sulfur Concentrations in Pulverized and Cleaned Coals

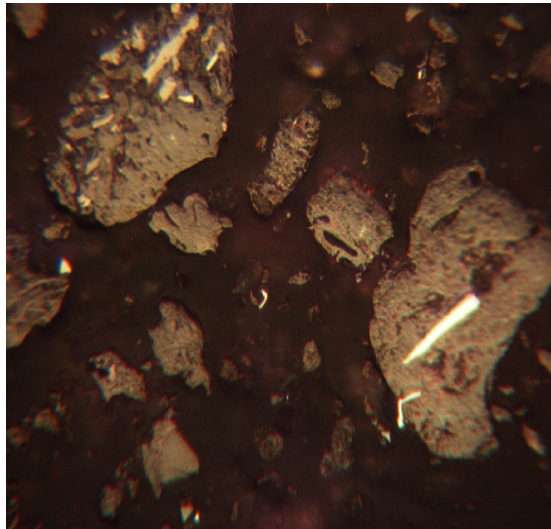


Figure 11. Photomicrograph (~ 450x) of the “cleaned” Wyodak coal.

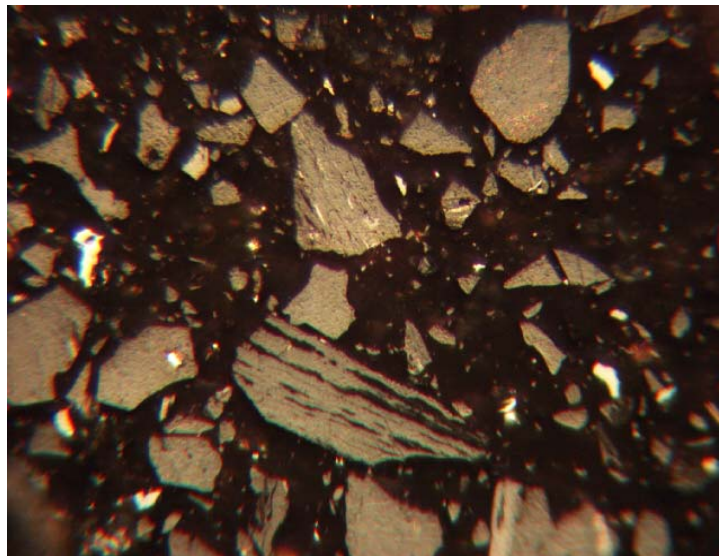


Figure 12. Photomicrograph (~ 450x) of “cleaned” (liberated and separated) Illinois #5 coal. Note the presence of very fine pyrite particles that are fully liberated but not separated.

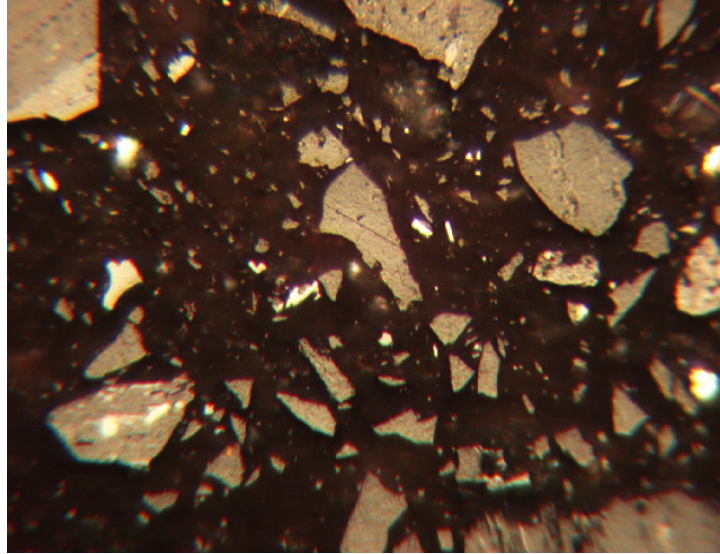


Figure 13. Photomicrograph of cleaned Pitt #8 coal, again showing the presence of very fine pyrite particles that are liberated but not separated.

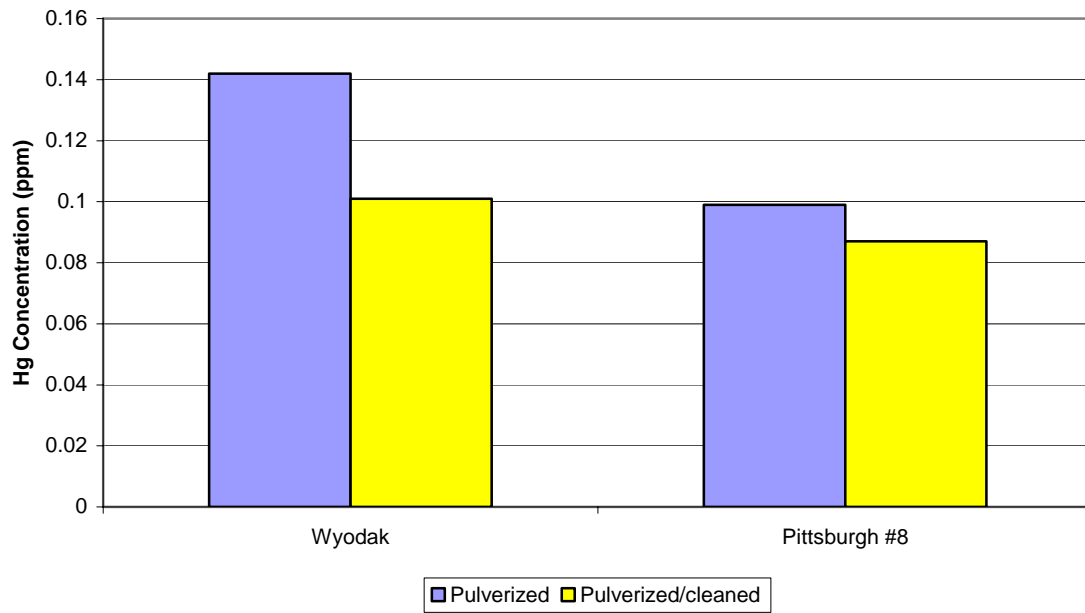


Figure 14. Mercury concentrations in Wyodak and Pittsburgh coals before and after removal of pyrite.

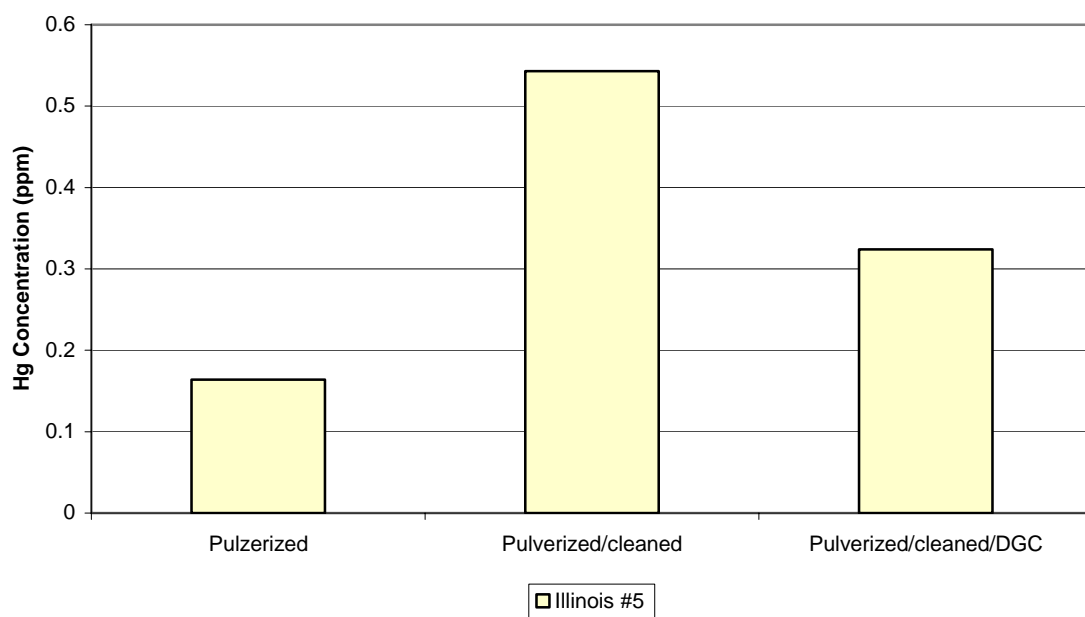


Figure 15. Mercury concentrations in Pulverized and cleaned Illinois #5 Coal

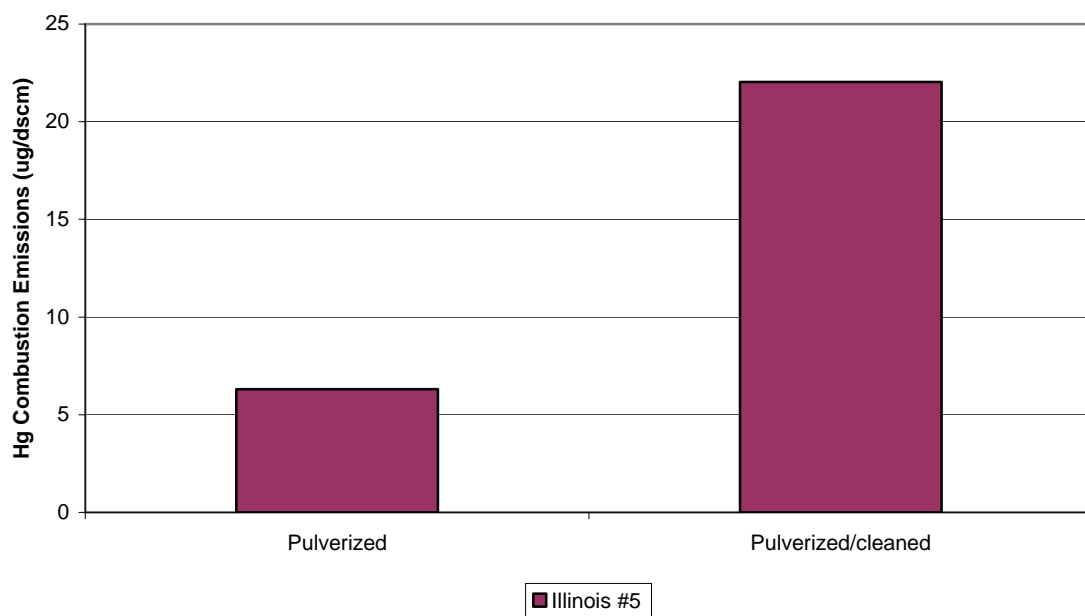


Figure 16. Total Mercury Emissions from Combustion Tests with Illinois #5 Coal

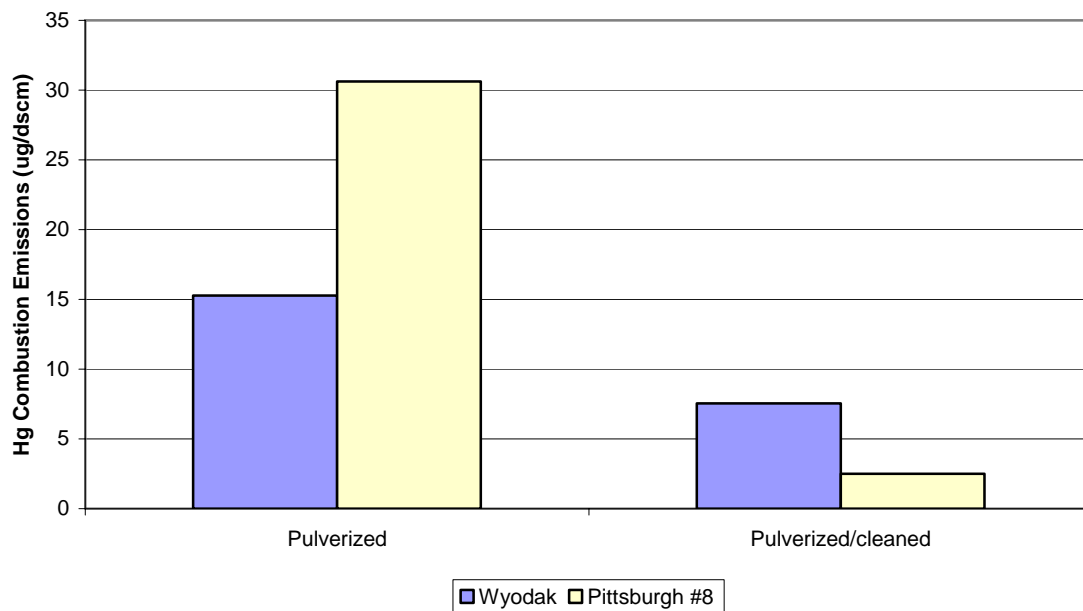


Figure 17. Total Mercury emissions during Combustion Tests with Wyodak and Pittsburgh Coals

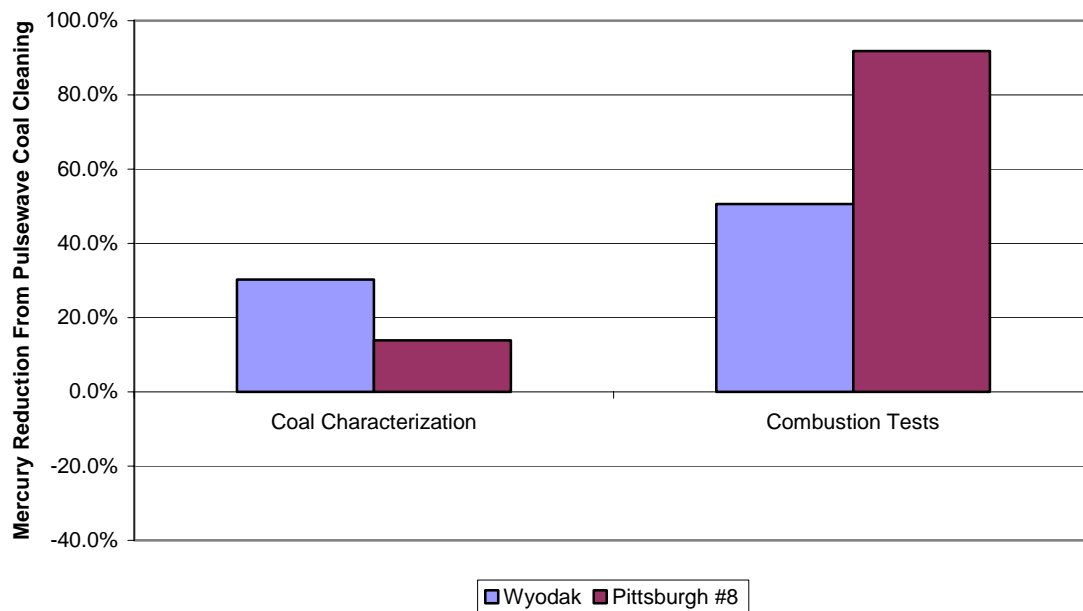


Figure 18. Comparison of Mercury Reduction from PulseWave Coal Cleaning measured from coal analyses and combustion tests.